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Title: REFLECTANCE SPECTROSCOPY SCREENING FOR ASBESTOS IN SOIL

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SYNOPSIS: A standardized method for spectral screening for asbestos in soil samples is described. Protocols for sample handling and analysis are provided.

Received by QA Unit:

REVIEWS:

TEAM MEMBER	SIGNATURE/TITLE	DATE
EPA Region 8	_____	_____
Syracuse Research Corp.	_____	_____

Revision	Date	Reason for Revision
1	1/7/00	Incorporated comments from USGS
2	7/11/00	Incorporated comments from USGS
3	3/28/02	Added description of QA requirements

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide a standardized method for analyzing soil samples for asbestiform mineral occurrence, to be used by employees of USEPA Region 8, or contractors/subcontractors supporting USEPA Region 8 projects and tasks.

This technique will be used as a screening tool to identify samples that contain asbestiform minerals. The spectral identification of asbestos-form minerals is limited by the analytical instrumentation, the abundance of asbestos-form materials present in each sample, and the physico-chemical (e.g., composition, moisture content, grain size distribution) state of the material containing the asbestos-form minerals.

This SOP describes the equipment and operations used for analyzing asbestos in surface soils. Site-specific deviations from the procedures outlined in this document must be approved by the USEPA Region 8 On Scene Coordinator, or Science Support Coordinator prior to initiation of the analysis.

It should be noted that this procedure is continually being developed, and may be modified or revised as needed. Any revisions to this SOP will be provided in the final report.

2.0 RESPONSIBILITIES

The Project Leader (PL) may be an USEPA employee or contractor who is responsible for performing reflectance spectroscopy analysis of asbestos in soil. The PL is also responsible for checking all work performed and verifying that the work satisfies the specific tasks outlined by this SOP and the Project Plan. It is the responsibility of the PL to communicate with the Field Personnel regarding specific analysis objectives and anticipated situations that require any deviation from the Project Plan. It is also the responsibility of the PL to communicate the need for any deviations from the Project Plan with the appropriate USEPA Region 8 personnel (On Scene Coordinator or Science Support Coordinator).

All personnel performing reflectance spectroscopy analysis are responsible for adhering to the applicable tasks outlined in this procedure while analyzing samples.

3.0 EQUIPMENT

- Spectrometer - Various models are acceptable. However, the instrument must be able to detect spectral effects in the 0.4 - 2.5 micron wavelength region, at a resolution better than or equal to 13 nanometers bandpass and a sampling interval less than or equal to 13 nanometers.

- Gloves - for personal protection and to prevent cross-contamination of samples. May be plastic or latex. Disposable, powderless.

- Personal Protective Equipment - as specified in the contractor's Health and Safety Plan.

- Field notebook -used to record progress of analysis effort and record any problems and field observations.

- Three-ring binder book - to store necessary forms used to record and track samples and analysis results. Binders will contain sample logbook pages for tracking samples, and data analysis sheets that record reflectance spectroscopy results for each sample.

4.0 INSTRUMENTATION

A reflectance spectrometer will be used to identify asbestos-form minerals. This instrument must be able to detect spectral effects in the 0.4 - 2.45 micron wavelength region, at a bandpass and spectral wavelength sampling as specified in Section 3.0, using a light source that covers the spectral range. Reflectance data of the samples of interest are compared to spectral standards in the United States Geological Survey (USGS) spectral library (or equivalent), using continuum removal and feature strength analysis methods.

5.0 PRECISION AND ACCURACY

The precision and accuracy of spectrometers is determined by the calibration procedure and post-processing computer program selected by the operator. Precision and accuracy requirements should be determined prior to analyzing any samples. The procedure for optimizing resolution and decreasing signal noise and interference is described in the manufacturer's operating manual.

6.0 METHODOLOGY

Samples are analyzed using a general purpose spectroradiometer that measures either relative or absolute light energy as a function of wavelength. Spectral data for each sample is then compared to a library of spectra that have been generated for various minerals and mineral classes.

6.1 Sample Preparation

Reflectance spectroscopy requires no sample preparation. However, samples that are analyzed in a fixed-based laboratory (rather than in the field) may be dried and sieved prior to analysis. If sample preparation step is performed, drying and sieving will be performed according to SOP #ISSI-LIBBY-01.

6.2 Analytical Procedure

The specific steps in collecting reflectance measurements are outlined in the manufacturer's operating manual. The following general steps should be followed:

1. Turn on the light source, if used, and let warm up for a minimum of 30 minutes (alternate light source may be the sun if making outdoor measurements).
2. Turn on the instrument.
3. Turn on the computer.
4. At the prompt, enter the controlling software.
5. Enter experimental conditions (e.g., integration times, etc.)
6. Position the light source to illuminate the sample/reflectance standard.
7. Position the instrument probe to view the sample/reflectance standard that is being examined. The sample/reflectance standard must fill the field of view of the probe.
8. Optimize the unit for the lighting conditions, as described in the manufacturer's operating manual.
9. Viewing the reflectance standard, collect a white reference (e.g., Spectralon®) measurement. If the signal/noise ratio is not acceptable, increase the spectrum averaging, or integration time.
10. When all setup procedures are complete, and the unit is collecting spectra, save data as described in the manufacturer's operating manual.
11. Collect spectra of wavelength standards (e.g. as supplied by the manufacturer), and reference minerals appropriate to the project, such as a tremolite for asbestos form mineral identification.

12. Periodically collect Dark Current measurements and white reference measurements, as described in the manufacturer's operating manual.
13. All spectra must be post processed to remove spectral effects of the white reference standard, e.g. as described in Clark *et al.*, 1990.

6.3 Data Presentation

Sample results will include sample identification numbers, a copy of the spectrum generated for each sample, and a qualitative (e.g., presence or absence) and/or semi-quantitative (e.g., relative abundance) indication of asbestos-form minerals, depending on the requirements of the project.

6.4 Instrument Calibration and Standardization

All spectra must be fully corrected to absolute reflectance before any analysis can be performed. Reflectance measurements are made relative to a "white" reference material, such as Spectralon®. The reflectance of the standard must be removed from the sample spectra, as described in Clark *et al.*, 1990.

Reflectance is derived by the ratio of the sample to the reference material, and is usually performed automatically in most modern commercial spectrometers. Calibration procedures are described in the manufacturer's operating manual for both wavelength and intensity. The instrument must be calibrated at the beginning of each sample analysis run, and repeated according to the manufacturer's recommendation or when instrument drift is detected.

6.5 Quality Control

A quality assurance program implementing specific quality control measures is required, and must be established by each performing laboratory. Such a program must include at a minimum: initial and continuing calibration standards, provisions for duplicate analyses, and corrective action plans for out-of-control results.

7.0 PERSONAL HEALTH AND SAFETY

The contractor responsible for performing reflectance spectroscopy analysis must have a Health and Safety Plan in place prior to analyzing any samples. All personnel are required to follow the procedures described in the Health and Safety Plan.

8.0 SAMPLE LABELING AND DOCUMENTATION

Each sample must be labeled with a unique identification number, as described in the Project Plan. All samples must be logged into the sample logbook prior to analysis, and results must be recorded on the data analysis sheets. Sample log sheets, and data analysis sheets will be maintained in a three-ring binder.

In addition, a field notebook should be maintained by each individual or team that is analyzing samples. The following information must be recorded for each day that samples are analyzed.

1. date
2. time
3. personnel
4. calibration results, including what reference materials were used
5. identification numbers for each sample analyzed
6. descriptions of any deviations to the Project Plan or this SOP and the reason for the deviation

9.0 REFERENCES

Clark, R.N., T.V.V. King, M. Klejwa, G. Swayze, and N. Vergo, High Spectral Resolution Reflectance Spectroscopy of Minerals: *J. Geophys Res.* **95,** 12653-12680, 1990.

Clark, R.N., G.A. Swayze, A. Gallagher, T.V.V. King, and W.M. Calvin, The U. S. Geological Survey, Digital Spectral Library: Version 1: 0.2 to 3.0 μm, U. S. Geological Survey, Open File Report 93-592, , 1340 pages, 1993. Online at: http://speclab.cr.usgs.gov.

Clark, R.N. Chapter 1: Spectroscopy of Rocks and Minerals and Principles of Spectroscopy, *Manual of Remote Sensing*, (A.N. Rencz, ed.) John Wiley and Sons, New York, p 3-58, 1999. Online at: http://speclab.cr.usgs.gov.

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